Utilisation of raw oil shale as fine aggregate to replace natural sand in concrete: Microstructure, surface chemistry and macro properties

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ABSTRACT

This study set out to examine the unconventional use of raw oil shale (OS) as fine aggregate in concrete instead of its traditional utilisation as a pollutant energy source aiming to hinder the depletion of natural resources. Oil shale particles have been used to replace sand in concrete with 30 wt.-% and 50 wt.-% ratios. Part of the used OS particles was treated with silane prior to their addition in concrete. The fresh, mechanical, and durability properties, along with the micro-properties and surface chemistry of mixtures, were analysed by running the slump, compressive strength, permeability, scanning electron microscopy (SEM) and fourier transform infrared spectroscopy (FTIR) tests, respectively. Five concrete mixtures consisting of 30 samples with the size of 150 mm x 150 mm x 150 mm were prepared to serve the testing programme of the study. The results revealed the formation of a well-bonded interfacial transition zone between untreated OS particles and the cementitious matrix due to the presence of pozzolanic materials in OS. However, micro cracks and enlarged capillary pores were witnessed in treated OS mixtures along with a weak interfacial transition zone between the treated OS and the cementitious matrix. Moreover, the use of 30 wt.-% untreated OS reduced the water absorption of concrete by 9% (absorption rate of 9.5%), while other mixtures experienced an increase in their water absorption with a maximum increase of 42% when using 50 wt.-% treated OS (absorption rate of 10.7%). All mixtures attained a reduction in their compressive strength compared to control sample when employing treated and untreated OS in concrete, with a minimum reduction of 13% when using 30 wt.-% untreated OS (compressive strength of 26 MPa). The strength reduction in untreated OS mixtures is due to the formation of free CaO and free SiO_2, which promote concrete expansion. The interference between silane and kerogen in OS was the main reason behind the strength reduction in treated OS mixtures. However, this reduction in mixtures' strength is considered minimal, allowing for their potential use in different construction applications like rigid pavement.

1. Introduction

Oil shale (OS) is a sedimentary rock that can be employed as a fuel to operate thermal power plants for energy production [1]. Its main constituent is an organic matter called kerogen, which, when decomposed, produces liquid oil and combustible gas [2–4]. OS has a potential economic significance in over 30 countries around the world, like the USA, Russia, Estonia, China, and Jordan [5]. However, many OS-rich countries are not exploiting it due to its negative impact on the environment during its combustion to produce fuel, accompanied by the high production cost of this fuel [6,7]. Besides fuel production, by-products from the burning of OS can be employed in producing plastics, insecticides, rubbers, sulphur, and many other petrochemical products [8].

Moreover, some researchers investigated the potential of utilising oil shale ash (OSA) in construction by producing modified cementitious and

bituminous materials and stabilising the soil [5,6,9–12]. The presence of silica, alumina and lime in the chemical composition of OS and its by-products made them a feasible option to replace cement in concrete mixtures [5]. Replacing cement with OSA induced promising results in terms of mechanical properties, where most previous works reported an enhancement in the compressive strength associated with an optimum replacement percentage that ranges between 5 wt% and 40 wt% [5,6,13, 14]. This improvement in the compressive strength was attributed to the presence of CaO and SiO_2 in the OSA, which works on forming calcium hydroxide and calcium hydrosulfoaluminate during the hydration of the ash [13].

Despite the significant enhancement in the properties of concrete containing OSA, it has a negative impact on the environment during its production, which involves burning the raw OS [15,16]. This serious aspect drove many researchers, environmentalists, governments, and environmental agencies to call for banning oil and gas production from OS, which implicitly involves its by-products [16–19]. Moreover, using the raw OS for other purposes that do not entail its burning might be a safe option to exploit this natural resource without imposing harmful impacts on the environment. Besides the safe use of the raw OS, it can also help reduce the depletion of other natural resources that experience high consumer demand. Natural aggregates, including sand, are one of those resources subjected to high depletion from the construction sector, where over 30 billion tons of sand are used yearly in construction [20–23]. However, a comprehensive review of the literature did not find any relative research work regarding the employment of raw OS as aggregate in concrete. Moreover, most of the conducted work focused on implementing the OSA as a substitute to cement in concrete mixtures.

In this research work, raw OS was incorporated in concrete to partially replace sand by 30 wt% and 50 wt%. Part of the used OS was treated with Vinyl tris(2-methoxyethoxy) silane prior to its incorporation in concrete in an attempt to improve concrete's durability. The water absorption of OS is higher than sand particles and replacing sand with OS might affect the overall durability of concrete [24,25]. Accordingly, employing silane as a treatment to OS might reduce its absorption to water and enhance the durability of concrete. Moreover, the interaction mechanism and interfacial bonding between OS and the cementitious constituents of concrete were thoroughly investigated. The influence of OS on concrete's mechanical, fresh, and durability properties was also studied. The experimental plan and methodology of this study are presented in Fig. 1.

The novelty of this study can be ascribed to its focus on the eco-implementation of the raw OS as aggregate in concrete and attenuating the construction sector's intense pressure on conventional natural aggregates. Using raw OS as a substitute for sand might offer an environmentally friendly divergence from its traditional use as a pollutant energy source.

2. Materials and methods

2.1. Materials and sampling

CEM 42.5 N ordinary Portland cement (OPC) complying with the specifications of the British standard EN 196–1 [26], river sand with a maximum size of 2 mm, and coarse limestone aggregate with a maximum size of 20 mm were used in this study. Raw oil shale (OS) obtained from the Attarat Um Ghudran area, south of Jordan, and supplied by Attarat Mining Company (AMCO) was incorporated into concrete as a substitute for sand. The obtained oil shale was ground and sieved to a maximum particle size of 2 mm. The chemical composition of the used oil shale is illustrated in Table 1 [27].

Vinyl tris(2-methoxyethoxy) silane, an environmentally friendly hydrophobic material, was used to treat part of the raw OS. OS was soaked in silane for 48 h and then left to dry at room temperature (approximately $23 \pm 2\,°C$) prior to its incorporation into the mixture.

Five concrete mixtures were prepared following the procedure outlined in the British Standard BS 1881–125 [28]. The mixtures consist of a control mixture (CS), a mixture with 30 wt% untreated OS as a substitute for sand (UO-30), a mixture with 30 wt% silane-treated OS as a substitute for sand (TO-30), a mixture with 50 wt% untreated OS as a substitute for sand (UO-50), and a mixture with 50 wt% silane-treated OS as a substitute of sand (TO-50). The mix design used in this study is shown in Table 2.

30 samples with the size of 150 mm x 150 mm x 150 mm were cast

Table 1
Chemical composition of Attarat Um Ghudran's oil shale [27].

Chemical composition	Content (%)
SiO_2	20.6
TiO_2	0.08
Al_2O_3	2.06
Fe_2O_3	0.65
MnO	0.002
MgO	0.71
CaO	33.6
Na_2O	0.19
K_2O	0.26
P_2O_5	3
Cl	0.07
S	2.7
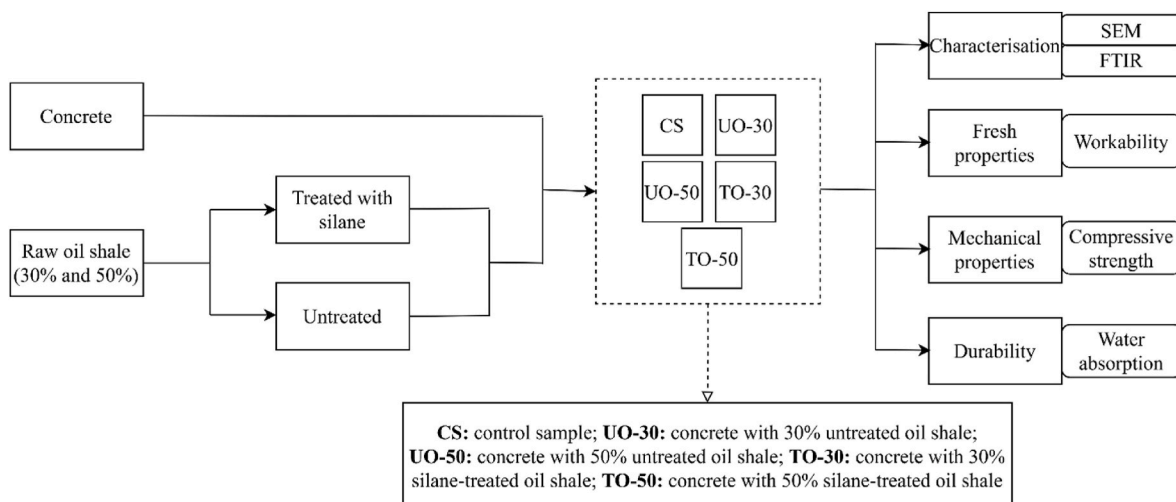
Fig. 1. Testing programme and mix formulations.

Table 2
Mix design of the used mixtures.

Component	Quantity (Kg/m³)
Cement (CEM 42.5 N; initial setting time $\geq$60 min, Cl $\leq$ 0.10, and SO$_3$ $\leq$ 3.5)	456
Coarse aggregate (a maximum size of 20 mm crushed limestone)	1073
Fine aggregate (a maximum size of 2 mm river sand)	661
Water	210

with the following arrangement: 6 samples for the CS mix, 6 samples for the UO-30 mix, 6 samples for the TO-30 mix, 6 samples for the UO-50 mix, and 6 samples for the TO-50 mix. All samples were cured in a water bath at 21 °C, after the removal of moulds, for 7 and 28 days.

2.2. Testing methods

2.2.1. Fresh properties
The slump test was used to evaluate the consistency of the produced mixtures in accordance with the BS EN 12350-2 guidelines [29–31].

2.2.2. Morphological analysis
A scanning electron microscope (SEM) equipped with energy-dispersive X-ray spectroscopy (EDS) was used to assess the changes in the mixtures' microstructure due to the addition of treated and untreated OS. Small fragments from the 28 days cured samples were gold coated and analysed using the Thermo scientific Phenom XL SEM.

2.2.3. Surface chemistry
Attenuated total reflectance-fourier transform infrared spectroscopy (ATR-FTIR) analysis was conducted on all mixtures to evaluate the changes in their functional groups and the interaction mechanism between OS and the cementitious composite. A PerkinElmer spectrum two FT-IR device equipped with ATR was used to serve this test. The analysis was run at a wavenumber range between 4000 cm^{-1} to 600 cm^{-1}.

2.2.4. Durability assessment
The long-term performance of all mixtures was investigated by running the water absorption test after 7 and 28 days of curing, following the procedure of ASTM C1585 [32–34]. After completing each curing period, the cube samples (150 mm x 150 mm x 150 mm) were dried in an oven at 100 °C until a constant mass was reached. Samples were submerged in water, and their masses were recorded after 2, 4, 6, 24, 26, 28, 30 and 48 h from soaking them in water. Moreover, the water absorption rate (%) for each mixture was calculated using the following formula:

$$Water\ Absorption\ (\%) = \frac{M_w - M_d}{M_d} X\ 100$$

Where, M$_d$ is the dry mass in grams and M$_w$ is the wet mass in grams.

2.2.5. Compressive strength
Controls AUTOMAX 5 50-C4652 testing machine was employed to assess the compressive strength of all mixtures after 7 and 28 days of curing following the BS EN 12390-5 guidelines [35–38]. A loading rate of 0.01 mm/s was used in this test. Three specimens were used for each mix to ensure reliability of the results.

3. Results and discussion

3.1. Fresh properties

The fresh properties of mixtures were evaluated in terms of their workability by carrying out the slump test. Results from this test are shown in Table 3. The presence of untreated OS within concrete reduced

Table 3
Workability of all mixtures.

Mixture	Slump value (mm)
CS	83
UO-30	57
UO-50	34
TO-30	96
TO-50	102

its workability, where a 59% and 31% drop in the slump value of UO-50 and UO-30 mixtures was observed, respectively, compared to control samples. This is due to the high water absorption of OS, which ranges between 10% and 12% [28] compared to river sand, that has a maximum absorption of 3% [25]. Accordingly, the raw OS will consume higher amounts of mixing water, which will reduce the workability of concrete. On the other hand, replacing sand with silane-treated OS increased the workability by 23% and 16% in TO-50 and TO-30 mixtures, respectively. OS was saturated with silane before its incorporation into concrete, which will create a hydrophobic film on the surface of OS. This hydrophobic film will seal the pores of OS, reduce its absorption, leaving more water in the mixtures and thus increasing workability.

3.2. Morphological analysis and interfacial bonding

The interfacial transition zone between OS particles and cement and the changes in the microstructure of mixtures were analysed by conducting the SEM analysis. As shown in Fig. 2a, the control mixture is distinguished by a highly dense structure with the absence of air voids, which denotes the high consistency of the mixture. Whereas replacing sand with untreated OS resulted in a less dense structure than control with the obvious presence of air voids within the cementitious matrix, as seen in Fig. 2b Those air voids could adversely affect the permeability of concrete and their strength. It is also evident from the micrographs that raw OS particles and the cementitious matrix developed a very well-bonded and intimate interfacial transition zone with the absence of delamination and micro cracks. This can be due to the comparable composition of the OS particles and sand (in terms of SiO$_2$ presence), and the inherent cementing properties of OS, where more than 55% of its composition are cementitious compounds (CaO, Al$_2$O$_3$, and SiO$_2$) **(Table 1)**, which enhance the bonding between hydrated cement and the surface of OS particles. This was interpreted by detecting numerous depositions of several hydration products, including C–S–H and Ca(OH)$_2$, on the surface of OS particles after running the EDS analysis.

On the other hand, the employment of silane as a treatment for OS has resulted in evident detachment between OS particles and the surrounding cementitious matrix (Fig. 2c). Additionally, micro cracks were observed within the cementitious matrix. This could be attributed to the interference between the applied silane and the inborn hydrocarbon kerogen compound present in OS, where both silane and kerogen possess hydrophobic natures, which impedes the bonding between the cementitious matrix and OS [39]. The presence of a silanol group in the applied silane might also increase the OS surface's smoothness, thus reducing the bond at the interfacial transition zone, i.e. lubrication effect as opposed to mechanical entanglement [40].

3.3. Functional groups and interaction mechanism

FTIR analysis was performed to determine the changes in the surface chemistry of the tested mixtures and the interaction mechanism between OS and the cementitious matrix. As illustrated in Fig. 3, some characteristic peaks can be observed in the spectra of the mixtures at 3747 cm^{-1}, 2359 cm^{-1}, 1410 cm^{-1}, 965 cm^{-1}, and 872 cm^{-1}. The 3747 cm^{-1} band is assigned to O–H stretching vibration bond, the 2359 cm^{-1} band is contributed to Si–H stretching bond, the 1410 cm^{-1} band is assigned to CH$_3$ and CH$_2$ asymmetric bending bonds, the 965 cm^{-1} band refers to
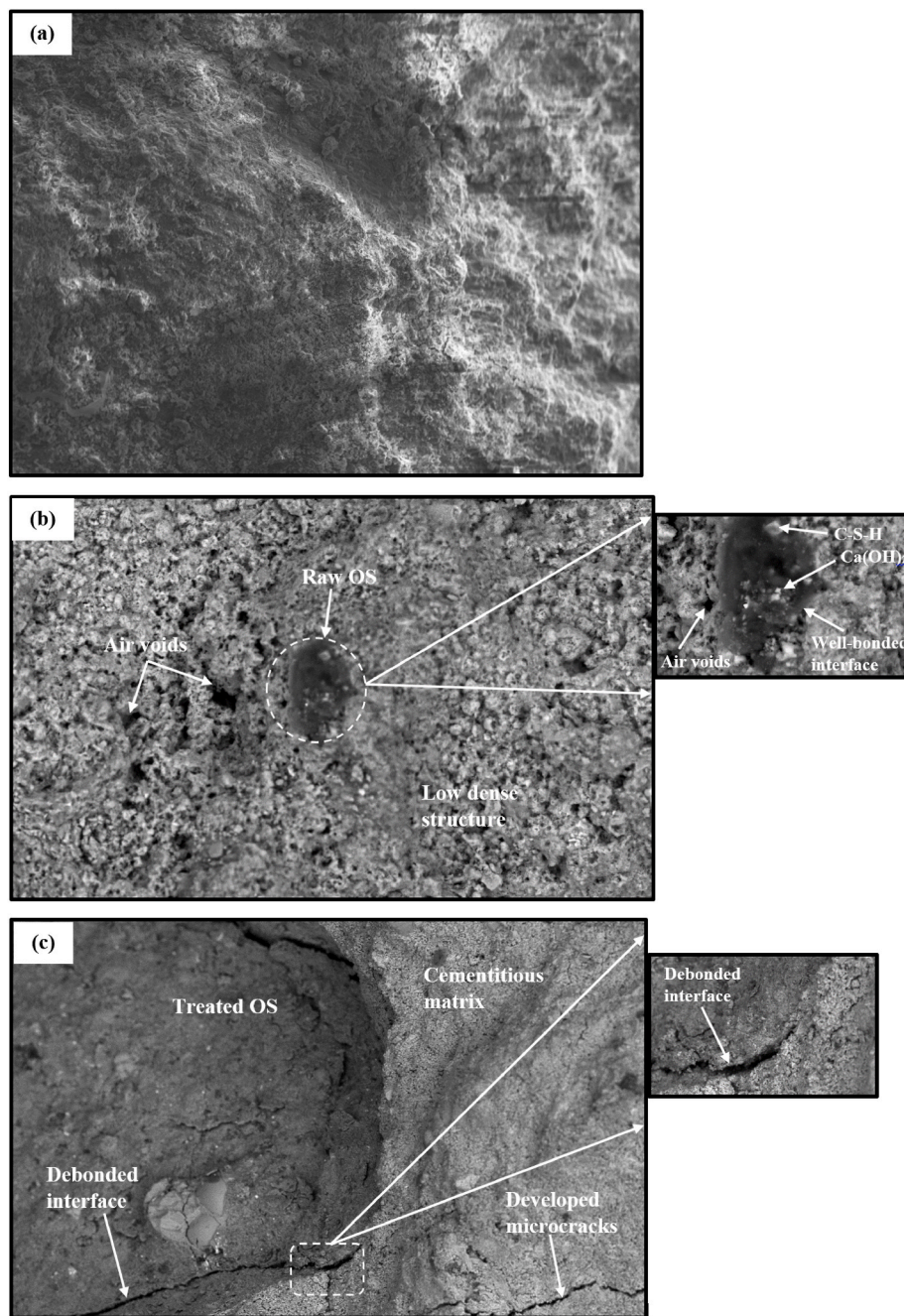
Fig. 2. SEM analysis of (a) control mixture, (b) concrete with untreated OS, and (c) concrete with treated OS.

Si–OH bonds, and the 872 cm^{-1} band is contributed to calcite (CaCO$_3$) [41–44].

As indicated in Fig. 3, the transmittance of the O–H bond can be seen to increase in concrete mixtures after the addition of treated OS, which indicates the presence of higher amounts of free water in this mixture compared to other mixtures. This might be due to the effect of silane in reducing the water absorption of OS, which leads to increasing the amount of free water in the mixture. The intensity of the Si–H bond was also observed to increase in concrete with treated OS, which denotes the presence of silicon deposits on the surface of OS as a result of the silane treatment. On the other hand, CH$_3$ and CH$_2$ asymmetric bending bonds were witnessed to have the highest transmittance in the mixture with raw (untreated) OS, which might refer to the benzene ring in the hydrocarbon kerogen compound that characterises OS [45]. The reduction in the intensity of those bonds in concrete with treated OS is related to

the influence of silane in covering the surface of OS, which reduces those bonds.

Interestingly, all mixtures were seen to attain high intensities of Si–OH and CaCO$_3$ bonds. Regarding the control mixture, the high intensity of those bonds denotes their abundance in natural sand and cement (more than 85% silica in sand and 65% lime in cement). Moreover, the incorporation of raw OS in concrete worked on increasing the intensity of Si–OH and CaCO$_3$ bonds, more than the control, due to the presence of high levels of SiO$_2$ and lime (over 55%) in OS teamed up with the silica and lime that already exist in sand and cement. On the other hand, concrete with treated OS is characterised by a sharp and robust Si–OH bond, which refers to the influence of silane in increasing this bond.
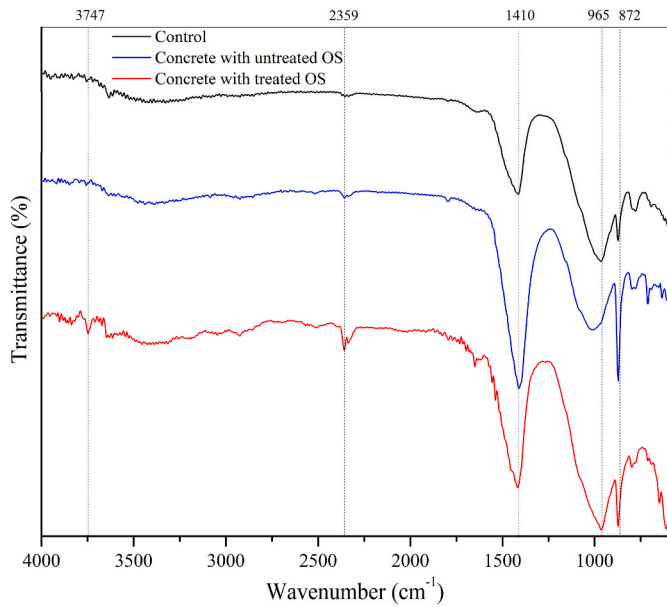
Fig. 3. FTIR analysis of concrete mixtures with OS.

### 3.4. Durability assessment through water absorption

The long-term performance of mixtures was studied by assessing their water absorption rate. As illustrated in Fig. 4, all mixtures modified with OS exhibited higher water absorption rates than control after 7 days of curing, where an increase of 13%, 12%, 28%, and 13% was observed in UO-30, UO-50, TO-30, and TO-50 mixtures, respectively. This might refer to the incomplete hydration process at 7 days, which results in the manifestation of air voids in mixtures and reduced bonding between OS particles and the cementitious matrix.

On the other hand, all mixtures demonstrated an increase in their water absorption rate after 28 days of curing except the UO-30 mixture, which attained a 9% reduction in water absorption compared to the control. Moreover, UO-50, TO-30, and TO-50 mixtures experienced 13%, 37%, and 42% increases in their water absorption rate, respectively. The superior performance of the UO-30 mixture could be attributed to its moderate content of CaO, Al_2O_3, and SiO_2 that enhance the pozzolanic reaction of OS with calcium hydroxide in cement and its bonding with the existent natural aggregates, which eliminates the formation of micro cracks within the mixture. Furthermore, the risk of expansion will not be an issue in this mixture since CaO and SiO_2 will be consumed during the hydration process due to their moderate content. Additionally, OS particles are characterised by their inherent hydrophobic nature owing to its main component, kerogen, which reduces their absorption to water. On the contrary, the increase in water absorption rate of the UO-50 mixture might refer to the high content of OS in concrete, which implies the presence of high levels of free CaO and free SiO_2 in the mixture resulting in increasing the volume of the capillary pores within the concrete which increases its demand to water [6]. Furthermore, free CaO might form excessive amounts of calcium hydroxide (Fig. 2b) after its hydration resulting in leaching and the formation of enlarged capillary pores and air voids, which would increase the water absorption of the mixture.

Nonetheless, the high water absorption rate of mixtures with treated OS (37% and 42%) could be attributed to the negative interaction between silane and kerogen in OS, as both are considered hydrophobic materials. The presence of kerogen in OS would reduce the absorption of OS particles to silane, leaving more silane on the surface of OS, which would negatively affect the bonding between OS and the cementitious matrix (evidenced in Fig. 2c).
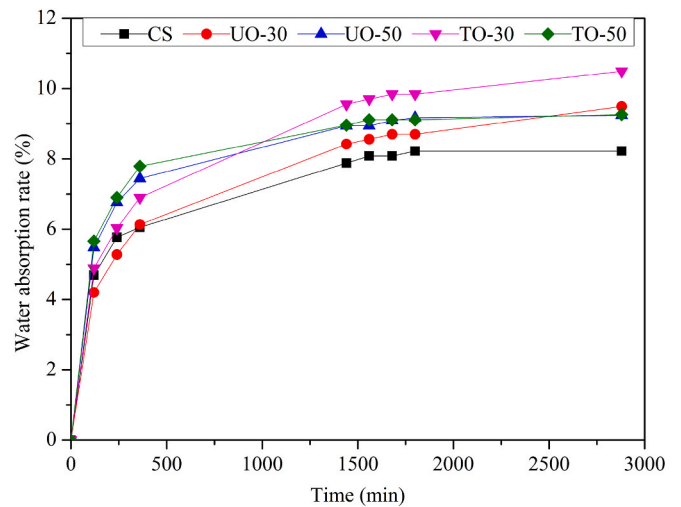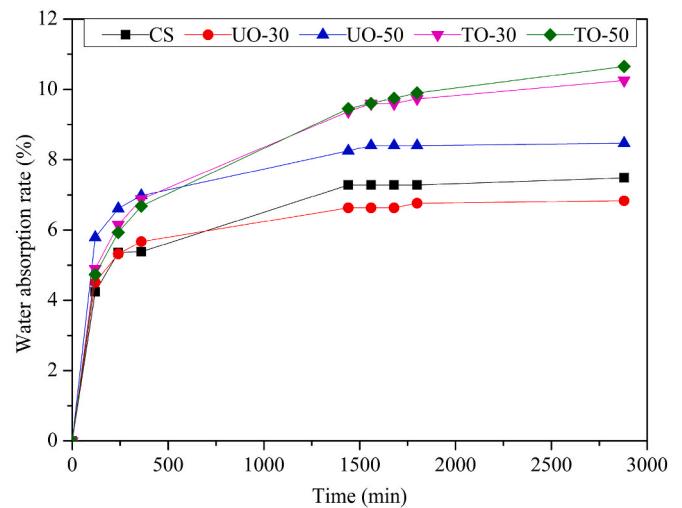
Fig. 4. Water absorption rate of concrete mixtures after: (a) 7 days and (b) 28 days of curing.

3.5. 3.5compressive strength

Fig. 5 shows the compressive strength results for all mixtures after 7 and 28 days of curing. It is evident from the figure that the control mixture possessed the highest compressive strength value at 7 and 28 days, while all the other mixtures, modified with OS, suffered from a drop in their strength. The compressive strength of UO-30, UO-50, TO-30, and TO-50 mixtures exhibited a drop compared to CS of 25%, 30%, 29%, and 31% at 7 days and 13%, 27%, 23%, and 26% at 28 days, respectively. In the case of mixtures containing the untreated OS, the reduction in the strength could be attributed to: (1) the presence of high free CaO and free SiO_2 content in concrete, which will prompt the expansion of the cementitious composite and the formation of cracks and air voids; (2) the formation of SO_3 in the mixture after the hydration of the surface of OS particles, resulting in forming calcium hydrosulfoaluminate, which works on enlarging the capillary pores of the concrete and promoting crack propagation [13]; (3) the poor interlocking between OS particles and natural aggregates and the cementitious composite due to the presence of hydrophobic kerogen on the surface of OS, which encourages slippage between particles when the mixture is exposed to loading.

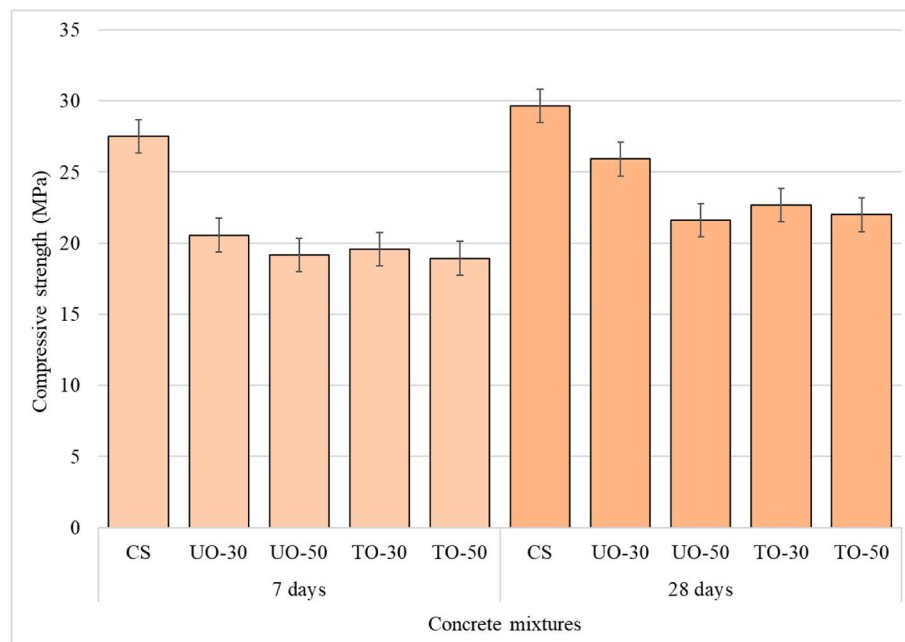On the other hand, the reduction of strength in mixtures with treated

Fig. 5. Compressive strength of concrete mixtures after 7 and 28 days of curing.

OS might refer to: (1) interference of silane with kerogen, where silane creates a loosely attached film on the surface of OS, which, in turn, reduces the bonding between OS particles and the cementitious composite (this was confirmed in the SEM analysis, Fig. 2c); (2) the presence of silane on the surface of OS, which promotes slippage between the particles and the cementitious composite; (3) the development of cracks and air voids in this mixture (as seen in Fig. 2c).

4. Conclusions

This research work aimed at assessing the use of oil shale (OS) particles in concrete mixtures as a replacement for sand. OS was employed in two forms; treated with silane and untreated (raw). The microproperties of mixtures and the interaction between OS particles and the cementitious matrix were investigated by conducting the SEM and FTIR analyses. The water absorbability, mechanical and fresh properties of mixtures were also studied. The results indicated that replacing sand with untreated OS would develop a well-bonded interfacial transition zone between OS particles and the cementitious matrix due to the high content of SiO_2 and pozzolanic materials in OS. On the contrary, replacing sand with treated OS promoted a detached interfacial transition zone between OS and the cementitious matrix owing to the interference between kerogen and silane. Furthermore, all mixtures exhibited a reduction in their compressive strength (minimum reduction of 13% when using 30 wt.-% untreated OS), with a higher reduction in samples containing treated OS (minimum reduction of 23% when using 30 wt.-% treated OS). This reduction in strength in treated OS mixtures is attributed to the presence of silane on the surface of OS, which promotes slippage between the particles and the cementitious composite. Moreover, the strength reduction in untreated OS mixtures is due to the presence of high free CaO and free SiO_2 content in concrete, which promotes the expansion of the cementitious composite. On the other hand, the use of 30 wt.-% untreated OS improved the impermeability of concrete (absorption rate of 9.5%), which could be due to the moderate content of CaO, Al_2O_3, and SiO_2 in this mixture. This would enhance the pozzolanic reaction of OS with calcium hydroxide in cement and its bonding with the existent natural aggregates, which reduced the formation of micro cracks within the mixture.

Credit author statement

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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